

Migration and Delivery of Filter Flavors

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INTRODUCTION

The current trend toward low tar delivery cigarettes has resulted in a concurrent decrease in the taste experienced by the smoker. Cigarette manufacturers are faced, therefore, with the problem of delivering adequate taste while maintaining low levels of tar and nicotine. There are several ways to increase cigarette flavor. Blending different tobacco types for increased flavor and adding flavorants to the tobacco are two techniques which are commonly used.¹ However, these methods possess the shortcoming that flavoring associated with the tobacco is altered and reduced as it traverses the filter segment.² Another technique for enhancing cigarette taste involves incorporating flavor additives into the filter, where they can be eluted into the smoke aerosol as it passes through the filter. Relatively high flavor delivery efficiencies can frequently be achieved in this manner despite low tar deliveries. Although this flavoring technique is currently recognized by cigarette manufacturers,³ very little information describing the behavior of filter flavors exists. This study addresses several fundamental questions relating to interactions between flavorant and filter and how such interactions ultimately affect the efficiency with which a filter flavor is delivered to the smoker.

EXPERIMENTAL

Experimental filter cigarettes were prepared by using conditioned 65-mm blended tobacco rods and 20-mm flavored filter tips. Filter tips were fabricated from 2.9 den./fil, 41,000 total den. #10 filter tow and a triacetin solution containing 10 model flavor compounds at a concentration of 10 mg/mL each. Finished 20-mm filter tips weighed 148.5 ± 1.3 mg each and contained 10% triacetin by weight and 0.134 mg of each of the 10 model "flavors." The following chemicals obtained from Eastman Organic Chemicals and from Pfaltz and Bauer, Inc., were used as model flavor compounds: cinnamyl isovalerate, cinnamyl propionate, 4-ethoxyacetophenone, eugenol, isoamyl benzoate, isoamyl cinnamate, isoamyl isovalerate, isoamyl phenylacetate, methyl benzoate, and methyl cinnamate.

Test cigarettes with an average wet TPM delivery of 21.4 mg were all prepared on the same day by attaching the flavored filter tips to 85-mm blended tobacco columns and stored in sealed glass jars. Cigarettes were sampled initially and 1, 2, 4, 6, and 8 wk after preparation for migration and delivery levels. Migration was determined by separating the filter tips from the tobacco rods and

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extracting and analyzing each for its flavor content. Flavor delivery was determined by analyzing the flavor content of an extract of particulates collected on Cambridge filter pads during smoking. A series of ventilated cigarettes were also prepared at 25 and 50% dilution, and their delivery was measured as a function of time.

The quantitative measurement of model flavor compounds was achieved by extracting filters, tobacco columns, or Cambridge pads with a solution of absolute ethanol containing 0.020 mg/mL decylaldehyde obtained from Aldrich as an internal standard. Complete extraction was demonstrated by spiking samples with a known amount of flavored triacetin and then analyzing the sample after 24 hr. Analysis of flavors present in the extracts was performed by gas chromatography on a temperature programmed 30-m WCOT OV101 capillary column. A Carlo Erba Fractovap series 2900 GC equipped with flame ionization detector (FID) was used in the splitless injection mode for the analysis. Peak height ratios with the internal standard were used for the measurement based on a seven-point calibration curve for each model compound. Each extract was chromatographed twice and represented a group of five experimental cigarettes. Four such groups of five cigarettes were measured to obtain each migration and delivery data point.

RESULTS AND DISCUSSION

Efficiency of Flavor Application

Table 1 lists the percentages of each flavor in freshly fabricated filter tips relative to the amount of flavor in the plasticizer. A flavor in plasticizer solution which is quantitatively applied by the brush applicator has an application efficiency of 100%. However, Table 1 shows that significant losses have occurred for isoamyl isovalerate and methyl benzoate. These two compounds are more volatile than the other additives and are more readily vaporized from the high-surface-area mist of plasticizer droplets generated by the brush applicator. The other less volatile compounds are not lost in this manner and are applied in virtually quantitative amounts. In general, it appears that compounds which boil below 250°C are not quantitatively applied by a brush applicator because of evaporation from the plasticizer mist.

TABLE 1
EFFICIENCY OF BRUSH APPLICATOR FOR FILTER FLAVORS

Flavor	Boiling Point, °C	Application Efficiency, %
Isoamyl isovalerate	193	51 ± 3
Methyl benzoate	200	81 ± 2
Eugenol	253	100 ± 6
Methyl cinnamate	254	88 ± 3
Isoamyl benzoate	262	92 ± 2
Isoamyl phenylacetate	268	92 ± 3
4-Ethoxyacetophenone	269	94 ± 3
Cinnamyl propionate	289	94 ± 3
Isoamyl cinnamate	298	105 ± 12
Cinnamyl isovalerate	313	95 ± 7
Average of last 8		95 ± 5

Migration of Filter Flavors

Table 2 shows the percentage of the total amount of flavor compound applied to the filter which migrated to the tobacco rod for six flavors as a function of cigarette age. Migration is not reported for the other four flavors because low migration and interferences prohibited their measurement in the tobacco column. However, for those compounds measured, it was observed that the flavor originally placed on the filter was quantitatively recovered from the tobacco and filter. Table 2 shows that migration took place most rapidly during the first week after the test cigarettes were

TABLE 2
MIGRATION OF FILTER FLAVORS WITH TIME

	Percent of Total Flavor Migrated to Tobacco				
	Wk 1	Wk 2	Wk 4	Wk 6	Wk 8
Isoamyl isovalerate	67	67	66	65	64
Methyl benzoate	20	24	28	28	31
Methyl cinnamate	10	15	18	23	22
4-Ethoxyacetophenone	6.1	7.9	9.9	12	14
Cinnamyl propionate	4.7	5.8	6.0	8.7	8.1
Cinnamyl isovalerate	3.8	7.8	9.2	19	16

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fabricated and that the rate of migration decreased with increasing cigarette age. After some time, equilibrium migration was achieved, and no further migration took place. The rate at which equilibrium was achieved and its final value varied from flavor to flavor and, therefore, must be determined largely by the properties of the flavor compounds.

The observed migration appeared to follow an exponential time dependence in which the physical limitations of no migration at week zero and attainment of equilibrium migration at some length of time were obeyed. The following model was, therefore, evaluated to describe flavor migration:

$$M(T) = M_{E0} (1 - e^{-kT})$$

where $M(T)$ is the migration at time T , M_{E0} is the equilibrium migration, e is the natural base, and k is the first order rate constant with units of reciprocal time. The model was fit to the experimental data by using nonlinear least squares regression analysis. The time dependence predicted by the model is shown as solid lines through the experimental data points in Figure 1. Figure 1 also shows some menthol migration data from an earlier study.⁴ The excellent agreement between the experimental data and the theoretical model supports the model's validity.

FIGURE 1
MIGRATION OF FLAVORS

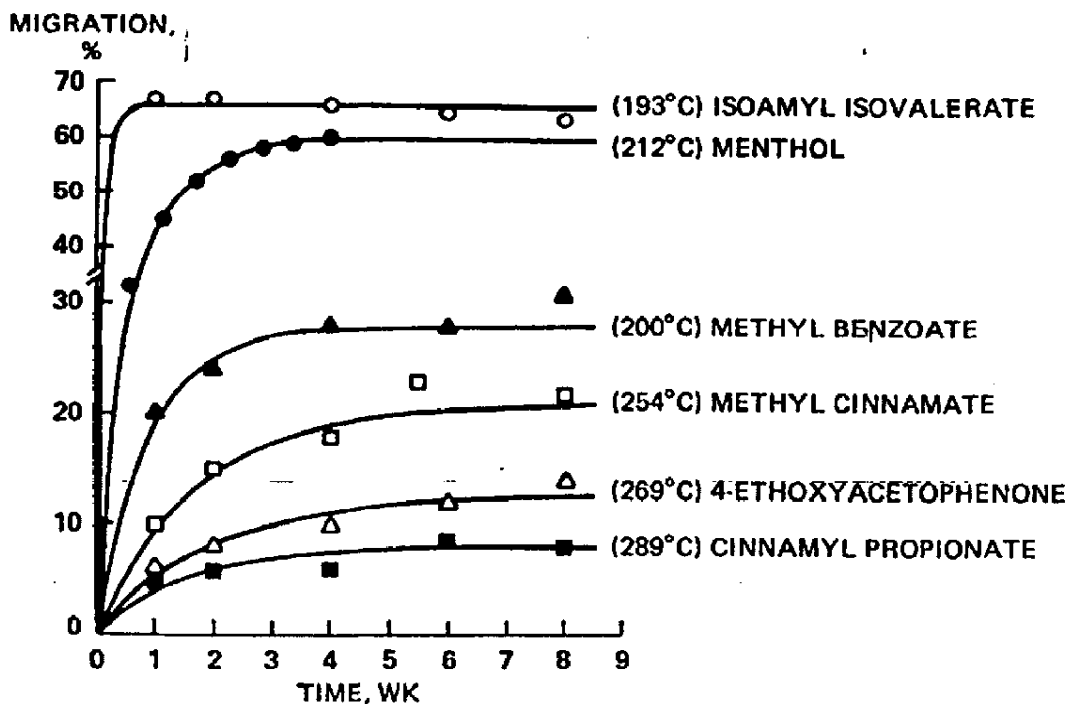


Table 3 lists the values of k and M_{EQ} determined from the model by nonlinear regression analysis. Values of $T_{1/2}$, which is the time required for migration to attain half its equilibrium value, are also given, since $T_{1/2}$ is physically more meaningful than k .

Values for the flavor migration rate constant, k , correlated with the volatility of the flavor in triacetin. Volatility is defined as the product of the vapor pressure of the pure compound times an activity coefficient which corrects the vapor pressure of the compound for its thermodynamic solubility and concentration in solution.

TABLE 3
KINETICS OF FILTER FLAVOR MIGRATION

Flavor	$\gamma_{25}^{0.01}$	P_{25}^0 (torr)	$\gamma_{25}^{0.01} P_{25}^0$ (torr)	k (wk ⁻¹)	$T_{1/2}$ (wk)	M_{EQ} (%)
Isoamyl isovalerate	6.72	0.9	6.05	8.98 ±.01	0.077	66 ± 2
Methyl benzoate	1.05	0.4	0.42	1.18 ±.15	0.59	28 ± 2
Methyl cinnamate	0.79	0.26	0.021	0.60 ±.27	1.2	21 ± 2
4-Ethoxyacetophenone	1.39	0.004	0.0056	0.51 ±.38	1.4	13 ± 2
Cinnamyl propionate	0.97	0.002	0.0019	0.68 ±.40	1.0	8 ± 1
Cinnamyl isovalerate	1.36	0.001	0.0014	0.33 ±.33	2.1	16 ± 5
Menthol	10.4	0.1	1.04	1.20 ±.10	0.58	60 ± 1

Values of the activity coefficients for the model compounds studied were determined at 25°C for a solute mole fraction of 0.01 by using the UNIFAC method.⁵ The linear relationship discovered between k and flavor volatility is described by the following equation:

$$k = 0.53 + 1.4 \gamma_{25}^{0.01} P_{25}^0$$

where γ is the activity coefficient and P^0 is the vapor pressure of the pure compound. A correlation coefficient of 0.9994 was calculated for the compounds studied, thus asserting the strong dependence of flavor volatility on migration kinetics. The data for menthol migration was not included in the analysis because filter menthol levels used to obtain that data were many times

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higher than the filter flavor levels used in this study. These findings imply a vapor phase mechanism for flavor migration in which compounds placed on the filter vaporize and diffuse to the tobacco rod, where they condense. This model, however, cannot predict M_{EO} , which is a thermodynamic quantity controlled by the relative affinity of the flavor for the tobacco versus the plasticized filler.

Delivery of Filter Flavors

The percentages of the total amount of each model compound delivered in the Cambridge pad particulates during smoking are listed in Table 4 as a function of cigarette age. For each flavor studied, with the exception of methyl benzoate, the delivery was most efficient at week zero and later decreased with time. Decreases in delivery efficiency were always largest during the first week, with delivery efficiency usually reaching a constant within experimental variance after about a month.

TABLE 4
DELIVERY OF FILTER FLAVORS

Flavor	Percent of Total Flavor Delivered					
	Wk 0	Wk 1	Wk 2	Wk 4	Wk 6	Wk 8
Isoamyl isovalerate	40 ± 5	20	20	21	22	17
Methyl benzoate	11 ± 2	7.2	10	11	12	13
Methyl cinnamate	33 ± 2	13	13	12	14	13
Triacetin ^(a)	9.1 ± .6	8.5	7.7	6.8	7.0	7.6
Isoamyl benzoate	55 ± 5	47	42	36	34	35
Isoamyl phenylacetate	51 ± 5	40	36	27	26	26
4-Ethoxyacetophenone	23 ± 2	11	8.7	7.4	8.2	10
Cinnamyl propionate	27 ± 3	15	12	9.5	9.6	9.9
Isoamyl cinnamate	29 ± 6	21	20	13	14	21
Cinnamyl isovalerate	31 ± 5	26	23	16	16	18

^(a)Present in filter as plasticizer at a concentration of 14.9 mg/20-mm tip.

Although the observed delivery efficiency decrease is in part attributable to migration to the tobacco column and subsequent losses to sidestream smoke and pyrolysis, Table 5 shows that migration accounts for only a small fraction of the observed delivery efficiency loss. This table shows that for every flavor for which the migration is known, with the exception of isoamyl isovalerate, the delivery efficiency loss after one week was much greater than the observed migration. In some cases, even though 95% of the flavor remained on the filter, about 50% of the delivery efficiency was lost. This is strong evidence that some process in the filter is acting to make the flavor compounds less accessible for elution into the smoke aerosol. To elucidate the mechanistic aspects of this process, the dependence of the rate of delivery efficiency loss was examined as it varied with the properties of the model flavor compounds. First order rate constants were calculated for the rate of delivery efficiency loss of each flavor by using nonlinear least squares regression analysis to fit an empirical model to the experimental data. The model used for this calculation was:

$$D(T) = D_0 e^{-kT} + D_{E0}$$

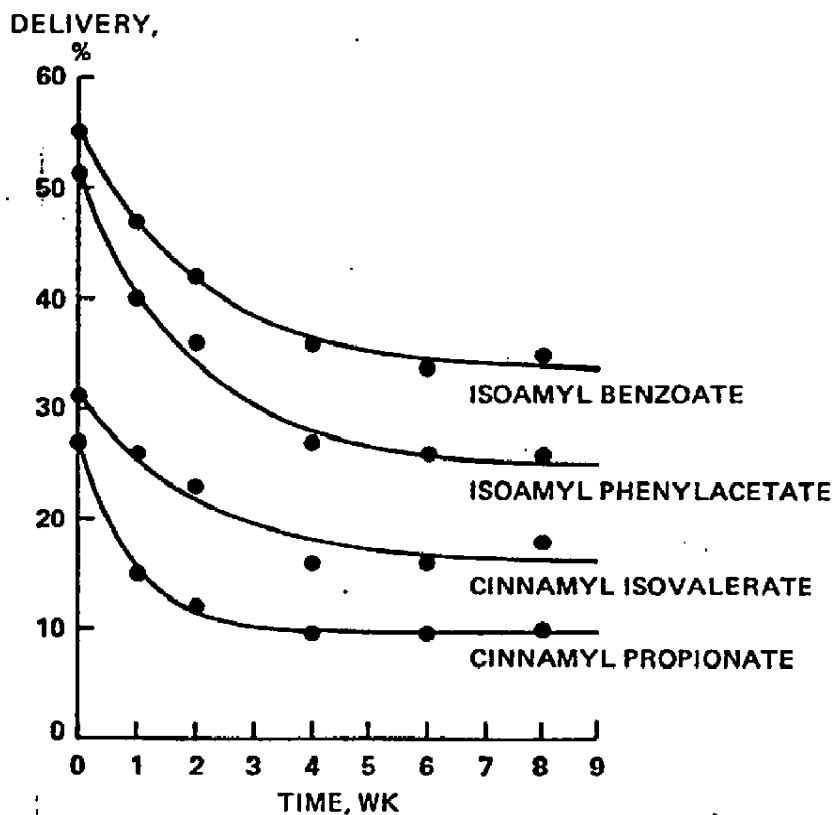
where $D(T)$ is the percentage delivered at time T , D_0 is the change in percentage delivery between time zero and equilibrium, D_{E0} is the percentage delivery at equilibrium, e is the natural base, and k is the rate constant for delivery efficiency loss. The validity of this model for the estimation of rate constants is demonstrated in Figure 2, in which the solid lines represent values of delivery efficiency predicted by the model from a fit to the displayed data points. Calculated values of k , D_0 , and D_{E0} are listed in Table 6. Values of $T_{1/2}$, the time required for half of the total delivery efficiency loss to occur, have again been reported to better demonstrate the time scale of this phenomenon.

TABLE 5
COMPARISON OF MIGRATION AND DELIVERY EFFICIENCY LOSS AT WEEK 1

Flavor	Migration, % of Total	Del Eff Loss, % Loss Ref to Week 0
Isoamyl isovalerate	67	50
Methyl benzoate	20	35
Methyl cinnamate	10	61
4-Ethoxyacetophenone	6.1	52
Cinnamyl propionate	4.7	44
Cinnamyl isovalerate	3.8	16

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FIGURE 2
DELIVERY OF FLAVORS



The values calculated for k , the rate constant for delivery efficiency loss, were found to correlate closely with the polar solubilities of the flavor compounds according to the following relationship:

$$K = -4.8 + 1.5 S_p; r = 0.97$$

Values of the polar solubilities are shown in Table 6 and were calculated by using a group contribution method. This empirical correlation indicates that solubility considerations control the rate at which materials placed on a filter in plasticizer solution become inaccessible for elution into the smoke aerosol.

These observations support a plausible hypothesis developed to account for the delivery efficiency loss phenomenon. It is known that after fabrication, several changes occur in the physical

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properties of cellulose acetate filters. These changes have been attributed to diffusion of triacetin into the filter fibers and include a lowered equilibrium water content and increased fiber density. These changes are, as well, most evident during the first week or two after filter fabrication, which is about the same time interval as observed for the delivery efficiency loss. Since flavors are applied to filters as a solution in triacetin, it is reasonable to assume that the flavors will also diffuse into the filter fibers. At week zero, the freshly applied triacetin and flavors are near the surface of the fibers and are easily eluted. As the filter ages, the plasticizer and flavors diffuse toward the cores of the fibers and no longer readily elute into the smoke aerosol. Flavors which are very soluble in triacetin diffuse into the fibers readily. However, flavors with lower solubilities diffuse into the fibers more slowly and to a lesser extent and, therefore, exhibit a slower rate of delivery efficiency loss.

TABLE 6
KINETICS OF DELIVERY EFFICIENCY LOSS

Flavor	k (wk ⁻¹)	T _{1/2} (wk)	D ₀ (%)	D _{EO} (%)	S _p
4-Ethoxyacetophenone	1.79 ±1.6	0.56	14.5	8.5	6.0
Cinnamyl propionate	1.13 ±0.3	0.89	17.3	9.7	5.2
Isoamyl benzoate	0.49 ±0.2	2.0	21.5	33.7	5.0
Isoamyl cinnamate	0.53 ±1.0	1.9	16.2	12.6	4.8
Cinnamyl isovalerate	0.49 ±0.5	2.0	15.5	16.0	4.8
Isoamyl phenylacetate	0.50 ±0.25	2.0	26.1	24.8	4.8

Effect of Filter Ventilation on Flavor Delivery

Tables 7 and 8 list the ratios of each flavor delivered by vented cigarettes versus nonvented cigarettes as a function of cigarette age for test samples prepared at 25 and 50% dilution. Expressing flavor delivery as a ratio relative to the delivery of the nonvented cigarette de-emphasizes the effects of migration and diffusion into filter fibers and exaggerates the influence of ventilation on the results. If the two volatile compounds tested, isoamyl isovalerate and methyl benzoate, are disregarded, it appears that ventilation has a relatively uniform effect on delivery and an average delivery ratio can be calculated for a ventilated cigarette as a function of time.

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These values, as well as delivery ratio values for triacetin and wet TPM, are summarized in Table 9. Although the data have significant variability, it is apparent from Table 9 that ventilated cigarettes deliver filter flavors more efficiently than they deliver TPM during the period immediately following their fabrication. However, the relative efficiency of flavor elution decreases with time such that, after two or three weeks, filter flavors are delivered less efficiently than tar relative to a nonvented cigarette. Expressing flavor delivery as a ratio should have removed the direct effect of flavor diffusion into the filter fibers on delivery, but the time dependence of the data strongly indicate that this mechanism is responsible for the observed delivery efficiency loss. Addition of ventilation must, therefore, exaggerate the effect of flavor diffusion into filter fibers on flavor elution. It is possible that the lower smoke velocity in the filter caused by ventilation results in a change in flavor elution kinetics such that diffusion out of the filter fibers becomes rate limiting. However, until conclusive experiments are performed, this rationalization of the data is only speculative.

TABLE 7
EFFECT OF VENTILATION ON FLAVOR DELIVERY AT 25% DILUTION

Flavor	Ratio of Flavor Delivered (Vented vs. Nonvented)					
	Wk 0	Wk 1	Wk 2	Wk 4	Wk 6	Wk 8
Isoamyl isovalerate	0.46	—	0.45	0.52	0.92	0.97
Methyl benzoate	0.82	0.86	0.74	0.90	1.08	0.85
Triacetin	0.89	0.79	0.81	0.62	0.66	0.60
Eugenol	0.89	1.06	0.92	0.82	0.88	0.88
Methyl cinnamate	0.86	0.91	0.85	0.81	1.00	0.86
Isoamyl benzoate	0.92	0.94	0.81	0.57	0.67	0.58
Isoamyl phenylacetate	0.90	0.92	0.82	0.57	0.76	0.65
4-Ethoxyacetophenone	0.91	0.80	0.80	0.68	0.86	0.81
Cinnamyl propionate	0.82	1.20	0.88	0.71	0.97	0.71
Isoamyl cinnamate	0.90	0.77	0.71	0.85	1.00	0.49
Cinnamyl isovalerate	0.89	0.76	0.76	0.66	0.92	0.51
Average last nine	0.89 ±0.03	0.91 ±0.15	0.82 ±0.06	0.70 ±0.11	0.86 ±0.13	0.68 ±0.15

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TABLE 8
EFFECT OF VENTILATION ON FLAVOR DELIVERY AT 50% DILUTION

Flavor	Ratio of Flavor Delivered (Vented vs. Nonvented)					
	Wk 0	Wk 1	Wk 2	Wk 4	Wk 6	Wk 8
Isoamyl isovalerate	0.57	0.61	0.47	0.50	0.62	0.51
Methyl benzoate	0.33	0.40	0.35	0.33	0.45	0.40
Triacetin	0.62	0.53	0.55	0.49	0.46	0.36
Eugenol	0.65	0.70	0.62	0.58	0.47	0.43
Methyl cinnamate	0.63	0.58	0.52	0.52	0.50	0.48
Isoamyl benzoate	0.68	0.64	0.59	0.46	0.42	0.34
Isoamyl phenylacetate	0.72	0.55	0.60	0.38	0.53	0.38
4-Ethoxyacetophenone	0.64	0.47	0.48	0.58	0.50	0.44
Cinnamyl propionate	0.63	0.46	0.62	0.55	0.81	0.47
Isoamyl cinnamate	0.68	0.46	0.47	0.52	0.58	0.30
Cinnamyl isovalerate	0.67	0.42	0.44	0.52	0.71	0.31
Average last nine	0.66 ±0.03	0.53 ±0.09	0.54 ±0.07	0.51 ±0.06	0.55 ±0.13	0.39 ±0.07

TABLE 9
EFFECT OF TIME AND VENTILATION ON AVERAGE FLAVOR DELIVERY

Time, wk	Ratio of Delivery (Vented vs. Nonvented)					
	25% Dilution			50% Dilution		
	Flavor	Triacetin	Wet TPM	Flavor	Triacetin	Wet TPM
0	0.89 ±0.03	0.89 ±0.09	0.81 ^(a)	0.66 ±0.03	0.62 ±0.09	0.56 ^(b)
1	0.91 ±0.15	0.79 ±0.18	0.83	0.53 ±0.09	0.53 ±0.12	0.55
2	0.82 ±0.06	0.81 ±0.09	0.76	0.54 ±0.07	0.55 ±0.11	0.54
4	0.70 ±0.11	0.62 ±0.03	0.76	0.51 ±0.06	0.49 ±0.04	0.53
6	0.86 ±0.13	0.66 ±0.06	0.84	0.55 ±0.13	0.46 ±0.09	0.55
8	0.68 ±0.15	0.59 ±0.09	0.88	0.39 ±0.07	0.36 ±0.03	0.57

^(a)Dry TPM ratio equals 0.84.

^(b)Dry TPM ratio equals 0.63.

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SUMMARY

The migration and delivery of filter flavor agents were studied by dissolving 10 model flavor compounds in triacetin at 10 mg/mL each and fabricating cigarettes with flavored filter tips. The concentrations of these compounds in filters, tobacco, and smoke particulates collected on Cambridge filters were determined by capillary-column gas chromatography initially and after 1, 2, 4, 6, and 8 wk. Flavor migration was found to obey first-order kinetics, with the rate constant being determined by the vapor pressure of the flavor compound. Delivery of flavor compounds decreased with time at a greater rate than could be explained by flavor migration. The proposed explanation for this behavior is that the compounds diffuse into the cellulose acetate fibers with time and become less accessible for elution into the smoke aerosol. Model compounds with nonpolar groups exhibited higher deliveries and smaller delivery decreases with time. These compounds are less soluble than polar compounds in the plasticized cellulose acetate fibers and remain near the surface, where they are more readily eluted. Ventilation initially increases the ratio of flavor to dry TPM delivered, but this advantage is lost as the cigarette ages.

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